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Building 7060	Those Eligible To Read the Attached	-
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	Date Date 6/6/44 8/10/44	To Read the Attached Copy 1 M. S. Smith Copy 1 M. S. Smith Put. sign and date below Date Name Date 6/6/44 8/10/44

This document has been approved for release to the public by:

Dank Hamm 3/10/95
Technical Information Officer Date
ORNIL Site

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#1370

and 3 figures.

CLINTON LABORATORIES

DATE June 14, 1944

o M. S. Smith

DEPARTMENT

FROM M. C. Leverett

DEPARTMENT

SECRE

IN RE: Off Gas Line - Building 7060

In connection with the chemical preparation being carried out by Mr. Coryell's section in Building 706C, my section has been asked to assist in some of the engineering work. It now appears that it will be desirable to install a pipe line to take the off gases from the dissolver set up in this operation and to discharge these gases into the duct which leads from the 205 building to the 205 stack. I would greatly appreciate it if you will undertake the supervision of the installation and laying out of this line since I believe that this is somewhat outside our normal range of activities.

The gases are at present drawn from the dissolver by means of an air jet which has sufficient capacity so that it can force the gases through a 2" line up the hill into the 205 duct without assistance from any other source. The volume of air flowing through this pipe line will be approximately 20 cu. ft/min. Preliminary calculations show that 2" schedule 40 iron pipe size pipe will be adequate and that approximately 550 ft. of this will be required. Mr. F. C. McCullough, who has been handling this work, advises me that approximately 600 ft. of 2" 18-8 S Cb pipe are in stock on the plant. It is desirable to use stainless steel pipe because of the possibly corrossive nature of the gases.

Attached herewith are copies of Mr. McCullough's sketches which I believe are self-explanatory. This piping should be complete and physically ready for use no later than June 25. This work can be done on the project which covers the chemical preparation mentioned above since it is necessitated by this preparation.

I shall be glad to supply any additional information which you may need and suggest that copies of any correspondence on this subject should be sent to Mr. Coryell. This memorandum confirms our telephone conversation of June 12.

ap

CC: Coryell
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Central File

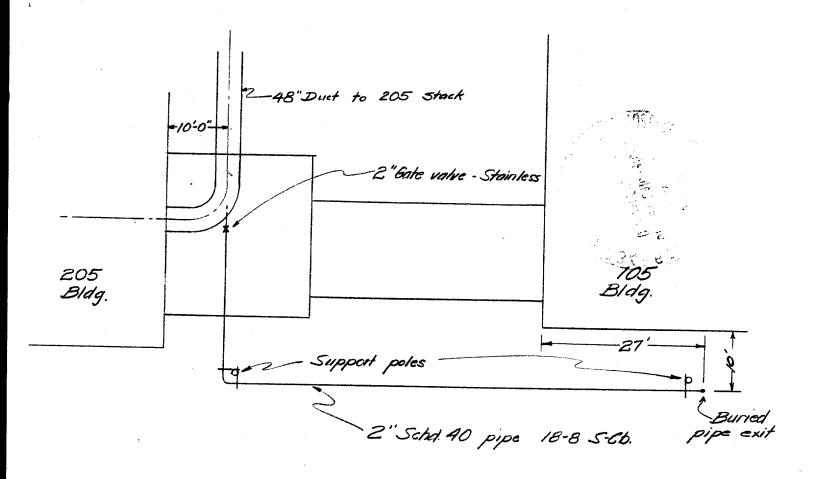
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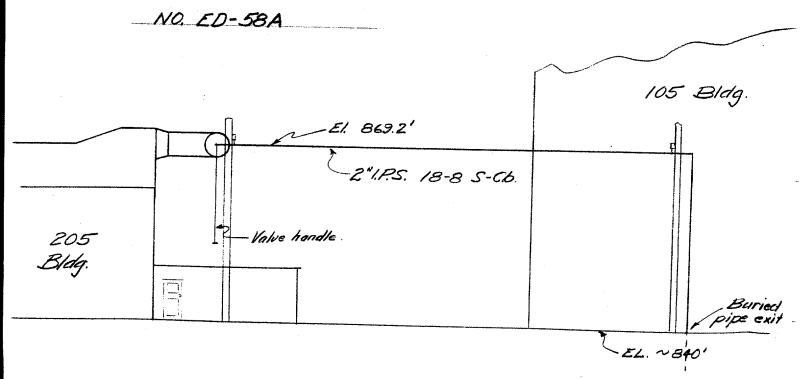
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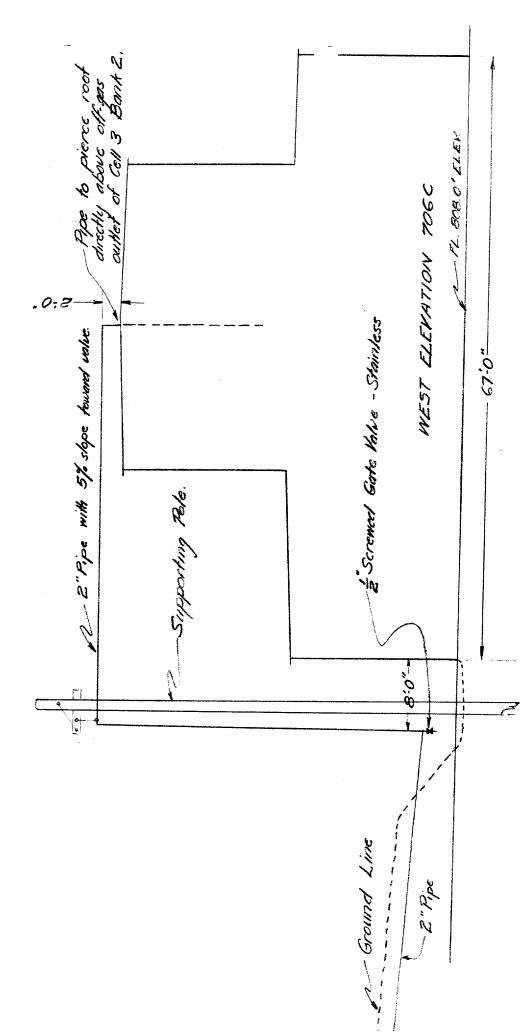
PLAN & ELEVATION OF OFF-GAS

LINE FROM 706-C to 205 STACK DUCT

Scale: - 16 = 1' June 13, 1944



Note:- Pipe duct from 706-C to 205 Exhaust duct to be 2" Sch. 40 18-8 S-Cb. All joints welded.

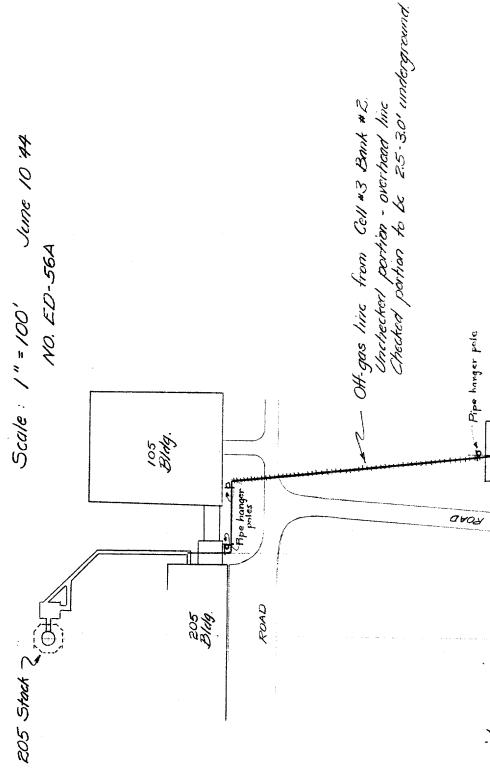


PROFILE OF OFF-GAS LINE Scale 3/32"=1' June 12, 1944 NO. ED-57A GELINE

BLDG. 706C to 205 STACK LAYOUT OF OFF GAS LINE from

DUCT

June 10 44 Scale 1"=100'



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Vertical -~100 ft.

706 C Bldg

Date



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Technical Information Office: Date Date

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J. E. Wirth, N.D.

H. C. Leverett

2 R. B. Brigge 5-6 L. C. Leverett
3 B. C. Rylen 7-8 R & C

DATE 1-31-5- COMINGENCE THE TWO STATES OF THE ACORD EDGE COMMISSIONS

205 Dissolver Off-Gan Monitoring

Unies, Deplassification Branch

I have done some more thinking about the question of monitoring the dissolver off-gas line in the 205 area after the shutdown of the 205 operations. As you know, it is planned that the 205 fan will be kept going in order to provide ventilation for 205 and 204, and to provide adequate dilution for gases from 706-C and 706-D introduced into the stack from the two-inch line up the hill.

I believe that the monitoring equipment is reliable within a factor of two or three for menon monitoring, and that it probably is giving results which are too high at present. The iodine monitoring equipment suffers from the fast that indine presumably condenses out in the sampling line, and it is therefore not known whether these results are reliable or not. If considerable quantities of indine were to continue to pass up the 205 stack, I think it would be necessary to continue to monitor the gases for lodine. However, in the case of 706-C, the off-gases from the dissolver (the principal source of iodine in the process) are passed through a scrubber counter-current to a stream of water. Laboratory tests indicate that the mater absorbs substantially all of the iodine so that very little of it is left in the gases that are discharged. This fact acts in opposition to the fact that the quantity of iodine released during the dissolving is comparable with or perhaps even greater than that released during a typical 205 dissolving. It is therefore likely that the gases discharged out the 205 stack will at their peak contain less active todine than has been present in the past from 205 dissolvings. In 706-D the dissolver off-gases will be scrubbed even more thoroughly, with a solution of MaCH, and it is therefore presumed that the same conclusion may be arrived at in the case of 706-D. The menon content of the games is of course little affected by the scrubbing.

In view of the foregoing comments I wonder if you would agree with me that it is unnecessary to attempt to improve the indine monitoring equipment, even though it is in a rather unsatisfactory condition at the present? I think that as a matter of operating routine we will want to record both indine and menon activities in the off-gas line whenever a fibrolying is made in 705-C or 705-D. In the case of the indine, this probably has only the values of determining that the indine concentration has not succeed by a large factor. In the case of the menon, the activities measured should have more or less absolute significance.

I would appreciate your letting me know if in your opinion saything beyond this is required.

CLASSIFICATION CANCELLED Jed David 3/8/95 ADD signature Date		2-202) as	te Disp
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incuments was authorized by DUE Office of	This accument consists	of 2	_	R. L. Dean
Assitingtion manne of August 22, 1994	pages and o	figures.	-	W. C. Jehnsen - H. S. Brown
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And the second to		•	,	J. E. Wirth
By Authority Of DOC			7.	H. A. Levy L. B. Emlet
By EE/8 Date AUG	<u> 23 1</u> 971	42%		B. C. Nylen Reading File
		▼		Central File

To: Mr. J. P. Sinclair

From: B. C. Nylen



This southend has been approved for relet to the public by:

David R. Hamm 3/9/4
Technical Information Officer Date

Off-Gas Tie-In Resulting in Near Spill in 706-0

We have reviewed the circumstances leading up to the near spill of active solution in 706-C on February 10 as presented in H. A. levy's letter of February 13 to W. C. Johnson. Additional facts bearing on the incident have also been developed which are of primary interest from the standpoint of preventing a recurrance of the mishap or of similar mishaps within the plant.

As pointed out in H. A. Levy's proposals, direct arrangement should have been made between 200 Area and 706-C supervision for the tie-in work between the 706-C off-gas line and 200 dissolver off-gas line. In this case 200 Area supervision relied upon two other groups to affect arrangements with 706-C supervision. This reflects no discredit upon the ether groups involved since real efforts were made by them to insure that the job be done safely. Final arrangements to do the work were made by C. W. Harp and included telephone agreement with E. L. Brady on February 7 or 8 to the effect that the neutralization of the storage tank at 706-C could and would be delayed until the morning of February 12 to permit completion of the tie-in work prior to that date. Two hours before the work was to be started en February 10 C. W. Harp informed R. L. Garber of the fact that the jeb was scheduled for a specific time and requested that he notify everyone concerned. H. A. Levy has stated that R. E. Garber had neither the necessary authority ner knowledge of 706-C operations to give clearance for this work, however, C. W. Harp points out that 90% of all repair orders are issued by R. E. Garber and practically all contacts with Maintenance are made by him for the 706-C group. Misunderstandings between 200 supervision and the Technical Division representative further the conclusion that direct and detailed plans should have been made between the operating groups involved.

The 100 Area supervision assumed responsibility on February 12 for operation of the steam syphon in the off-gas line located within the 200 Area. The exact details of jet operation are being settled between L. B. Emlet and H. A. Levy, however, in recognition of the proposals made by the latter to prevent reoccurance of the situation the following steps have been taken:



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- 1. The off-gas valve in the line has been chained and locked in open position. The key has been given to H. A. Levy and full control of the valve will be in the hands of 706-C supervision.
- 2. The valve controlling eff-gas steam jet pressure has also been chained and the key given to 100 Area supervision. We understand all valve adjustments will be made by them to maintain negative pressures as designated by 706-C supervision.
- 3. Any future maintenance work involving the off-gas line between 706-C and the 200 Area stack will be carried out only after arrangements have been made and a member of 706-C supervision is in presence to supervise the necessary work.

We suggest that similar near accidents in the future be immediately brought to the attention of other departments involved in order that corrective steps may be taken at once to prevent reoccurance. In this instance the letter describing the incident was not received until four days later.

B. C. Nylen

Superintendent - 200 Area

BCN /maw

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pages and 0 figures.

No. 2 of 1 copies, Series 1

cc:>M. D. Peterson

W. A. Rodger

W. P. Bigler

A. C. Vallado

E. J. Witkowski

L. R. Michener

TO: File

40479

FROM: L. R. Michener

July 14, 1945

706-D OFF-GAS SYSTEMS, (REVISED)

Two off-gas systems are installed in 706-D:

- 1. The A-4 scrubber system, which provides vacuum for A-1 and A-5 only.
- 2. The A-16 scrubber system, which provides vacuum for A6, A8, A9, B1, B3, B5 (15) B6, B12, B19, B20, B21

A-4 System

The recommended flow rate through A-4 is 160 divisions (4 G.P.M.) on flow meter. Cell differential pressure should be $\langle 0.5^n.$

To put all of A-4 vacuum on A-1, close valve A5 - A3 valve on PB-1.

To divide vacuum between Al and A5, make regulations with A5 - A5 valve on PB-1.

To obtain vacuum in either A-1 and/or A5, valve in AE-B9, PB-1, must be open. This is the header valve to 205 Bldg.

The A-4 line to 205 Bldg. has a valve in the pit outside Room 1. This valve is to remain open during operation of A-1 and A-5. The line continues underground to the Christmas tree behind 706-C, entering the system between the valve fartherest east and the next valve to the west of the first valve.

In Cell A, the A-4 off-gas line to 205 contains a 1/2" drain line, with valve, leading to Cell A ditch. There is no outside control. This valve is to be left slightly cracked. In case of a desire to drain this part of the line, close valve at 706-C Christmas tree and allow a reasonable amount of time for drainage.

DATE BY A TOTAL BASE OF COMMISSION CHIEf, Declassification Branch Chief, Declassification Branch

This document has been approved for release to the public by:

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July 14, 1945

A-16 System

The recommended flow rate thru A-16 is 10 divisions on flow meter (1 G.P.M.). Call differential pressure should be 7.5"

To open A-16 to small vacuum fans in fan house, or to 205 Bldg., extension handle valve in AE-Fl, first floor, must be open. This valve opens both cell A and Cell B lines to vacuum from fans.

A-16 can obtain vacuum from three sources:

- 1. The small fam outside fam house. This is the regular source. There is a drain, with valve from fam housing, which goes to W-11. The drain valve should be kept closed except when in use. There is also a drain valve from the bottom of the condensate trap located inside the fam house. This runs to W-11. The valve should be closed except when in use. These lines should be drained each shift during use of fam. The valve farthest east at Christmas tree must be closed during use of No. 1 fam.
- 2. The secondary small fan inside fan house. This is to be used in case of failure of No. 1 fan. In such case, suction line to No. 1 fan must be blanked off by maintenance and the valve to No. 2 fan.
- 5. The 205 Building system. In case of failure of No. 1 and No. 2, close valve to No. 2 fam, blank off No. 1 fam, open sunken valve two feet south of small fam house, open valve farthest east at 706-C Christmas tree, and open the remaining valves at the tree to cut in 205 Building.

There are three drains in the A-16 off-gas line between A-16 and fan house:

- 1. One of the two inside drains is controlled through AW-G12, first floor. This opens a $1/2^m$ drain to sump of Cell A. Leave closed except when draining.
- 2. A cyclone system for collection of condensate has been installed in the 4" discharge side of A-16 off-gas line and is located above A-6. The cyclone is drained by valve in a one inch line, controlled through AW-Kl2, first floor. Drainage goes to W-11. Drain once each shift.



3. The outside drain is located two feet from the north-east corner of 706-D and runs to W-11. A funnel and valve arrangement is provided at intersection of off-gas line and hot drain line for decontamination purposes. Leave closed except when necessary to drain.

A-6 can be cut into or out of the A-16 system by operation of valve in AE-B4, Second Floor. When vacuum is needed elsewhere, this valve should be closed unless vacuum is needed in A-6 for jetting.

A8 and A9 have individual off-gas valves on the third floor.

The one inch discharge line at the bottom of A-16 is provided with a valve (operated through third floor) for the purpose of filling A-16 with decontaminating agents. Leave open at all times during Run.

A system of introducing ammonia gas into the A-16 off-gas is in use for the purpose of neutralizing corrosive fumes. A gas cylinder is located on first floor near P-B.-3, feeding through regulating valves on second floor, near AW-Al2. A manometer equipped with equalizer is provided at this point. The ammonia traverses a 1/2" line to first floor and through AW-J13 to a point just above the main valve into A-16. Ammonia will thus be normally fed into the bottom of Al6. It is possible, however, to add ammonia to the discharge side of A-16 at a point in the off-gas line about 10 feet east of the exit from cell. Use 3" pressure on the NH 3 manometer and flush the gas in with 5# air (read on pressuge gage to left of NH₃ manometer.





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By	L. R. Michener	111	2
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This document has been approved for release to the public by:

Technical information Officer
ORNL Site

W. A. Rodger

W. P. Bigler

A. C. Vallado

E. J. Witkowski

L. R. Michener

7/6/45

To: File

From: L. R. Michener

This document co	nsists of 2
pages and	O figures
No. 1 Se of 6	copies, Series A

706D OFF-GAS SYSTEMS

file 1-5

Two off-gas systems are installed in 706D:

- 1. The A-4 scrubber system, which provides vacuum for A-1 and A5 only.
- 2. The A-16 scrubber system, which provides vacuum for A6, A8, A9, B1, B3, B5 (14), B6, B12, B19.

A-4 system

To put all of A-4 vacuum on A-1, close A5-A3 valve on PB-1.

To divide vacuum between Al and A5, make regulations with A5-A-3 valve on PB-1.

To obtain vacuum in either Al and/or A5, valve in AE-B9, behind PB-1, must be open. This is the header valve to 205 Building.

The A-4 line to 205 Building has a valve in the pit outside Room 1. This value is to remain open during operation of Al and A5. The line continues underground to the Christmas tree behind 706-C, entering the system between the valve farthest east and the next valve to the west of the first valve.

The recommended flow rate through A-4 is 160. Cell differential pressure should be >0.5.

The recommended flow rate through A-16 is 70. Cell differential pressure in Cell B should be >0.5ⁿ.

A-16 System

To open A-16 to small vacuum fans in fan house, or to 205 Building, extension handle valve in AE-Fl must be open. This valve opens vacuum to fans from both cell A and cell B.

A-16 can obtain vacuum from three sources:

1. The small fan outside fan house. This is the regular source. There is a drain, with valve, from fan housing, which goes to hot drain line. The drain valve should be kept closed except when in use. There is also a drain valve from the bottom of the condensate trap located inside the fan house. This runs to Wil. The valve should be closed except when in use.

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For The Atomic Energy Commission

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- 2. The secondary small fan inside fan house. This is to be used in case of failure of No. 1. In such case, suction line to No. 1 fan must be blanked off by maintenance and the valve to No. 2 fan opened.
- 3. The 205 Building System. In case of failure of No. 1 and 2, close valve to No. 2 fan, blank off No. 1 fan, open sunken valve two feet sough of small fan house, open valve farthest east at 706-C Christmas tree, and open the remaining valves at the tree to cut in 205 Building. Under normal use of No. 1, or even No. 2, the valve farthest east at the Christmas tree will remain closed.

There are two drains in the A-16 off-gas line between A-16 and fan house:

- 1. For draining inside cell A, open valve in AW-G12, first floor. This opens a 1/2" drain to sump of cell A.
- 2. The drain for the outside is located two feet from the northeast corner of 706-D and runs to 706-C hot drain tank. A funnel and valve arrangement is provided at intersection of off-gas line and hot drain line for decontamination purposes.

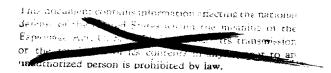
A6 can be cut into or out of A-16 system by operation of valve in AE-B4, second floor. When vacuum is needed elsewhere, this valve should be closed unless needed for transfer of solution into A-6.

AS and A9 have individual off-gas valves on the third floor.

Ammonia System

A system of introducing ammonia gas into the A-16 off-gas is in use for the purpose of neutralizing corrosive fumes. A gas cylinder is located on first floor near PB-3, feeding through regulating valves on second floor, near AW-Al2. A manometer equipped with equalizer is provided at this point. The NH₃ traverses a 1/2" line around Cell A to a spot behind PB-1 where it drops through floor and into the A-16 off-gas discharge line leading to fan house.

L. R. Michener





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DATE: SUBJECT:	January 5, 1952 Dissolver Bibliography	ORNE CENTRAL FILES NUMBER 52.1-21
BY:	F. L. Culler	02.1 -
TO:	H. E. Goeller	(a. ·
Before res	ading this document, sign and date below:	

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6. F. L. Culler
7. Y. E. Coeller

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January 5, 1952

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By Authority Of De

Gr Changed To

Mr. H. E. Goeller

Oak Ridge National Laboratory

P. O. Box 6 Arco, Idaho

Subject: Disselver Bibliography

Dear Sir:

During my most recent trip to Idaho A. M. Rom requested that we obtain and forward all Unit Operations reports concerning dissolving procedures for NP, MTR, and EHR fuels.

The following is a list of reports issued on dissolving procedures applicable to the Idaho Chemical Process Plant.

Off-Gas Treatment

CF-51-10-55	Operation of Pure Gas Catalytic Oxidizer E. O. Nurmi to F. L. Culler
CF-51-4-33	Suggested Experimental Work for Removal of Oxygen and Oxides of Nitrogen from D.O.G. Streams W. L. Carter & J. M. Holmes to F. L. Culler
CF-51-7-11	Proposed Radioactive Gas Separations System for Pilot Plant W. H. Farrow Jr. to F. L. Steahly
CF-51-10-128	Off-Gas Processing Studies CT-32 E. P. Reichardt to F. L. Steahly
CF-51-9-148	Dissolver Off-Gas Treatment, Arco Quarterly Report 5-7, 1951 E. P. Reichardt to F. L. Steahly
FLS-692	Dissolver Off-Gas Treatment - Arco E. O. Nurmi to F. L. Steahly
CF-51-11-157 CF-51-9-135 CF-51-9-94 CF-51-9-47	UNOP Status Report 11-26-51 UNOP Status Report 9-26-51 UNOP Status Report 9-19-51 UNOP Status Report 9-12-51
CF-51-8-228	UNOP Status Report 8-30-51

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H. E. Goeller

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January 5, 1952

Off-Gas Treatment (Cont)

CF-51-8-52	UNOP Status Report 8-6-51	
CF-51-7-176	UNCP Status Report 7-30-5	L
FLS-777	UNOP Status Report 7-15-5	L
FL3-766	UNCP Status Report 7-2-51	
FLS-758	UNOP Status Report 6-25-5	L
FLS-745	UNCP Status Report 6-18-5	L
FLS-737	UMOP Status Report 6-11-5	L
FLS-725	UMOP Status Report 6-4-51	
FLS-713	UMCP Status Report 5-28-5	L
FL8-699	UNOP Status Report 5-21-5	L
FLS-653	UNOP Status Report 4-30-5	Ļ

CF-51-9-48

DOG Treatment - Arco

Quarterly Report, May 1951 - July 1951

E. P. Reichardt to F. L. Steahly

MIR

CF-51-7-140

Mitrie Acid Dissolution of MTR Assemblies

E. O. Hurmi, D. L. Foster

EBR

FLS-709

KHR - Quarterly Report, 2/10/51 - 5/10/51

E. M. Sampson, Jr.

CF-51-8-151

EER Slug Dissolving - Quarterly Report for

May through July, 1951

E. M. Sampson, Jr.

ORNL-1137

Dissolution of KHR Slugs

E. M. Sampson, Jr. (To be published soon)

MP Slugs

CF-50-10-44

Metallurgical Treatment of Alloy Slugs Versus

Dissolving Rate

A. T. Gresky, E. O. Nurmi

FIS-491

Quarterly Report, Dissolving of U-Al Alloy,

P-10 Type Slugs, 8/10/50 - 11/10/50

D. L. Foster, E. O. Nurmi

FLS-587

Quarterly Report, Dissolving of U-Al Alloy

J Type Slugs, 11/10/50 - 2/10/51

D. L. Foster, E. O. Nurmi



H. E. Goeller

_ 4 _

January 5, 1952

MP Slugs (Cont)

FLS-710 Quarterly Report, J-Slug Dissolving 2/10/51 - 5/10/51 D. L. Foster, E. O. Murmi CF-51-8-115 Quarterly Report, Dissolution of U-Al Alloy J Type Slugs, Fume Recovery, 5/10/51 - 7/10/51 D. L. Foster, E. O. Nurmi FLS-632 J-Slug Dissolver Operating Procedure Details E. O. Nurmi CREL-1195 Dissolution of U-Al Alloy Slugs D. L. Foster, E. O. Murmi (To be published soon) Metallurgical Factors Affecting the Dissolving *CF-50-12-23 Rate of 7.5% U - 92.5% Al Alloy

*Metallurgy Division Report

We will secure copies of each of these documents and forward them to you, with copies to A. M. Rom if possible, as soon as they can be obtained.

D. E. Hamby to J. H. Frye, Jr.

Very truly yours,

OAK RIDGE NATIONAL LABORATORY

F. L. Culler, Chief

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Process Design Section Chemical Technology Division

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X-426 (Revised 1-52)

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QUARTERLY REPORT - A Method for the Analysis of Nitrogen Tetroxide in Dissolver Off-Gases

TO:

F. L. Steahly

FROM:

L. E. Line, Jr.

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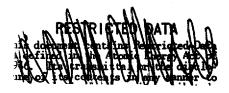
For: H. T. Bray, Supervisor Laboratory Records Sept.

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To: F. L. Steahly



Chemical Technology Division Laboratory Section

Report Period: 11/10/51-

2/10/52

Problem No. TDSI-

Part

QUARTERLY REPORT

Title: A Method for the Analysis of Nitrogen Tetroxide in Dissolver Off-Gases

Work by: L. E. Line

Secret Notebook No. 1406

1.0 Introduction

In connection with the study of gas clean-up in fumeless dissolving 1 , 2 , it became necessary to devise methods for analyzing a gas stream for smaller quantities of nitrous gases than could be determined by Orsat analysis. This memorandum describes a method for the analysis of $N_2O_4(NO_2)$ in concentrations ranging downward from 1 or 2 percent to less than 100 parts per million in the gas stream. Such a method will be applicable to NO, upon conversion of the latter to N_2O_4 .

The method herein reported has been developed only to the extent necessary for study of the scrubbing of the off-gas stream. For more general use, further study may be necessary.

2.0 Summary of the Method

The method is based on the reactions,

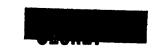
$$N_2O_4 \text{ (or 2NO}_2) + 2 \text{ NaOH} \longrightarrow \text{NaNO}_2 + \text{NaNO}_3 + \text{H}_2O$$
 (1)

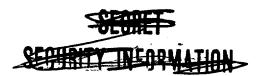
and

 NO_2^- + acetic acid + α -naphthylamine + sulfanilic acid \longrightarrow red azo dye (2).

The colorimetric reagent, sometimes called Griess Reagent, can be made sensitive to less than 0.01 ppm of nitrite in solution.







A measured volume of gas is passed successively through five gas washers of the fritted disk type, each containing 100 ml of 4M NaOH. The alkaline scrub in each washer is transferred quantitatively to a volumetric flask of the appropriate size and diluted to the mark with nitrite-free distilled water. Analyses are performed on solutions made by mixing aliquots of these solutions with a definite excess of acetic acid and a definite volume of Griess Reagent and diluting to the mark in another volumetric flask of appropriate size. After allowing 45 minutes for development of the pink color, transmittances are measured in a Beckman Model DU Spectrophotometer, using a wave length of 525 mm and a 1-cm. cell. Concentrations of nitrite are read from a previously established calibration curve obtained with standard nitrite solutions containing comparable amounts of acetic acid and sodium acetate. Percentages of N₂O₄(NO₂) in the gas sample are then calculated from the dilution factors, the volume of the sample, equation (1), and the ideal gas laws.

Beer's Law was found to hold in solutions up to 2 ppm of sodium nitrite. Solutions varying in acetic acid concentration from 4 to 8M but with (acetic acid)/(sodium acetate) fixed at about 15 gave points falling on the same straight line when - log I/Io was plotted against ppm of sodium nitrite.

When checked against weighed samples of N_2^{0} the method yielded 80% of the nitrite expected on the basis of equation (1). The procedure will probably give high results when NO is present. Sample data and calculations are presented.

3.0 Notes and Experimental Details

The use of Griess Reagent for detection and analysis of nitrites is not new^3 , and the method has received recent attention by Rider and Mellon 4 . It



seemed necessary, however, to check and extend the previous work in order to adapt the method to the analysis of nitrous gases. In particular, since the $N_2C_{\downarrow \downarrow}$ was to be absorbed in sodium hydroxide solution and the latter neutralized with acetic acid, a study of the effect of sodium acetate and acetic acid on color development seemed warranted.

3.1 Materials and Solutions

Griess Reagent was prepared by dissolving 1 g. of sulfanilic acid and 0.2 g. of α -naphthylamine in 600 ml of a 50% aqueous solution of acetic acid. The colorless solution was stored in an amber bottle.

Standard nitrite solution was prepared by dissolving 0.3000 g. reagent grade sodium nitrite in one liter of solution with nitrite-free water and diluting 10.00 ml of this solution to one liter in a volumetric flask. Such a solution contains 3 ppm of NaNO₂.

Other reagents, acetic acid, sodium hydroxide, sodium acetate, and distilled water, showed the absence of nitrite.

3.2 Transmittance Curve for a Neutral Nitrite Solution

Figure 1 shows that the maximum absorption for a 0.3 ppm neutral sodium nitrite solution occurs at a wave length of 525 mm. Accordingly, this wave length was employed for the analyses.

3.3 Time for Full Color Development and Color Stability

Figure 2 shows the variation of transmittance I/Io with time for solutions 0.12M in sodium acetate and 0.7 to 4M in acetic acid. These show (a) that



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the transmittance decreases as the ratio of the concentration of acetic acid to sodium acetate increases, (b) that the time for color development decreases with increasing concentration of acetic acid, and (c) that in each case the transmittance remains essentially constant after 45 minutes.

An idea of the stability of the colored solutions that had been left standing in diffuse light may be gained from Table I. The values of I/Io are essentially unchanged. For solution 6 the change in ppm corresponding to the change in I/Io is 0.08.

Table I

Stability of Color Formed with Nitrite Solutions and Griess Reagent

2M acetic acid; 0.12M sodium acetate; = 525 mm; 1 cm cell

Solution	ppm NaNO	I/Io after 45 minutes	I/Io after 2 - 4 hrs.	I/Io after 44 hrs.
1	0.16	0.834	0.807	0.820
2	.32	.694	.680	.693
3	.63	.475	.460	.474
4	•95	.322	.314	.325
5	1.27	.224	.216	.226
6	1.59	.152	.150	.161
7	1.74	.130	.126	.137

3.4 Calibration Curve

On the basis of the characteristics of transmittancy curves (not shown) with varying ratios of (acetic acid)/(sodium acetate) the ratio of about 15:1 was selected for the standard solutions. Investigation of a series of

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standard solutions ranging from 3.7 to 8M in acetic acid with the concentration of the latter fifteen times that of sodium acetate gave points falling on the same straight line when -log I/Io was plotted against ppm of NaNO₂, as may be seen from Figure 3. The lower line represents data for a series of solutions 2M in acetic acid and 0.12M in sodium acetate. One might include these data and draw the best straight line among the points if great accuracy is not required. It is of interest that the data follow Beer's Law in the range of concentration studied.

Thus, within limits, one has only to add to the aliquot of the scrub a quantity of acetic acid such that after neutralization and dilution to the appropriate volume (acetic acid)/(sodium acetate) = ca. 15.

With the spectrophotometer and a 1 cm cell as little as 0.01 ppm sodium nitrite in the solution can be detected. The sensitivity can be greatly increased by the use of longer columns of solution, such as may be obtained with Nessler tubes.

3.5 Effect of Nitrate Ion

Rider and Mellon have shown that nitrate ion present to the extent of 1000 times that of nitrite causes no interference.

3.6 Data and Calculations in a Run

Table II gives some data and calculations for purposes of illustration. A sample of gas that had been scrubbed by passing through a packed tower countercurrent to a stream of 20% KOH was submitted by T. S. McMillan and W. L. Johnson.

A sample of the influent gas was also submitted. Analyses by the method herein described show that 99% of the N_2O_4 was removed.

Table II

Illustrative Data and Calculations

Scrubber No.	Nitrite Concent Influent Gas (30	trations, ppm (100 ml soluti 03 cc) Effluent Gas (2	on) 65 cc)
1	75.0	0.47	
2	4.8	0.08	
3 (44)	2.0	0.05	
4	1.0	0.02	
5	0.9	<u>0.05</u>	
Totals	83.7	0.67	

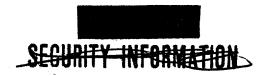
Sample Calculation for Influent Gas:

Mol. wt.
$$N_2O_4$$
 \longrightarrow $2NO_2$ is 80 at 25°
g N_2O_4 = $\frac{83.7 \times 10^{-6} \times 100 \times 92}{69} = 0.0111$
cc N_2O_4 = $\frac{nRT}{P}$ = $\frac{0.0111 \times 82 \times 300}{80} = 3.4$
% N_2O_4 = $\frac{3.4}{3.03} \times 100 = 1.1$

Similar calculation for effluent gasgives % $\rm N_2O_{l_4}$ = 0.011

3.7 Direct Check of the Method

While the method seems farily reliable for analysis of nitrite, a direct check against known amounts of $\rm N_2O_4$ seemed desirable. Some 80% of the expected



nitrite was found. While this result shows the method to be adequate for study of off-gas processing it leaves an error which exceeds that ordinarily involved in colorimetric work. The procedure is now being checked for errors in technique and for the possibility that not all N_2O_{\downarrow} reacts according to equation (1). The latter consideration is important in clean-up of N_2O_{\downarrow} , as the formation of other nitrous gases might create an additional problem.

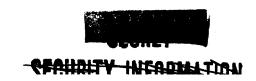
3.8 Effect of the Presence of NO

When NO is present the combination of NO $_2$ and NO is likely to give high results, as such mixtures are reported 5 to behave chemically as N $_2$ O $_3$ which reacts with water or alkali to yield two moles of nitrite instead of one.

4.0 References

- 1. Memo, L. G. Anderson to F. L. Steahly, CF 51-9-172, September 13, 1951.
- 2. Memo, Lloyd E. Line, Jr. to J. R. Flanary, October 19, 1951.
- L. M. Dennis, "Gas Analysis", The MacMillan Company, New York, 1925,
 p. 218.
- 4. B. F. Rider with M. G. Mellon, Anal. Chem., 18, 96(1946)
- 5. Yost and Russel, "Systematic Inorganic Chemistry", Prentice-Hall, New York, 1944.

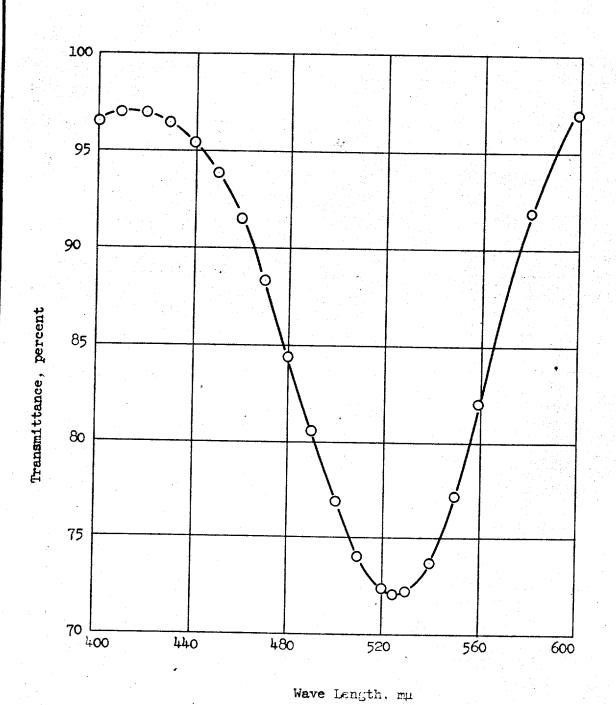
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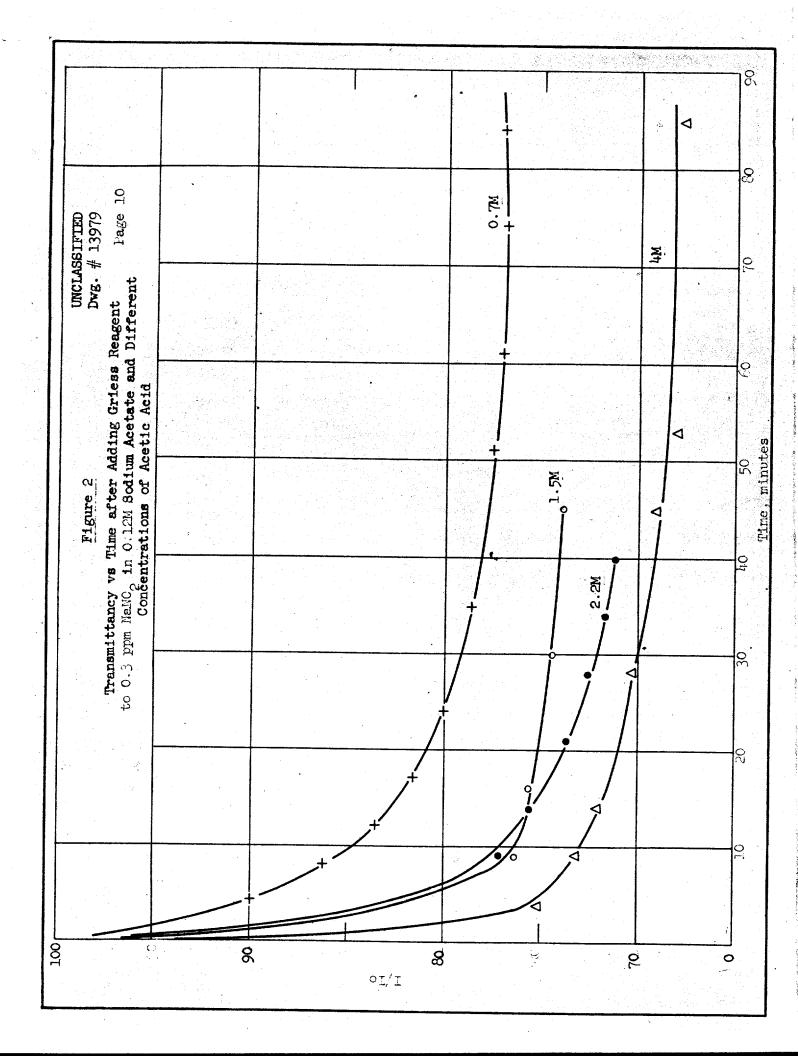


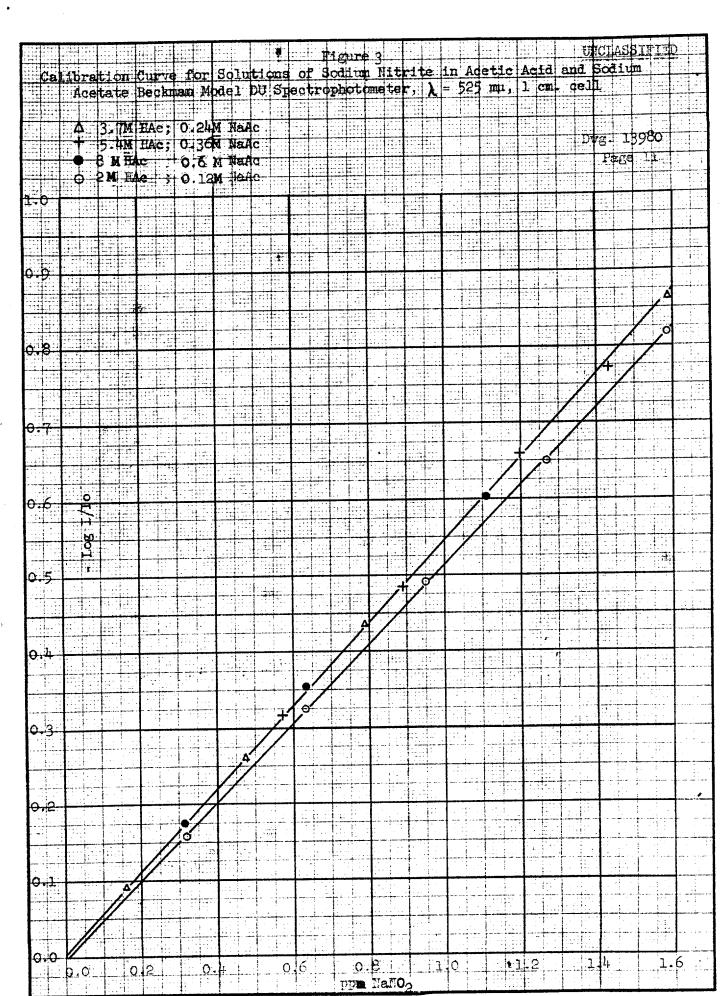
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Figure 1

Transmittance - Wave Length Curve
After Adding Griess Reagent to
0.3 ppm NaNO2 in Water









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POST OFFICE BOX P OAK RIDGE, TENNESSEE

DATE:

March 19, 1952

SUBJECT: Dissolver Off-Gas Processing - Quarterly Report for Period 11/10/51 to 2/10/52

TO:

F. L. Steahly

FROM:

T. S. McMillan

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TO: F. L. Steahly

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Chemical Technology Division Unit Operations Section

FROM: T. S. McMillan, Problem Leader

Report Period: 11/10/51 - 2/10/52

QUARTERLY REPORT

CT-32

Title: Dissolver Off-Gas Processing

Work By: T. S. McMillan, Problem Leader, E. O. Nurmi, Group Leader, W. L. Johnson

Secret Notebook No. 1412, 1801

1.0 Introduction

The purpose of this work is to develop a method of removing radioactive krypton and xenon from the off-gas generated during slug dissolution.

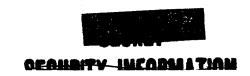
The contemplated procedure is to adsorb these elements upon Columbia CXA activated carbon at low temperature. However, before this operation can be performed, it is necessary to remove from the gas mixture all components which would interfere with the adsorption. Procedures for removal of I₂, NO, NO₂, and CO₂ have been described in previous reports. A procedure for removal of oxygen and N₂O by hydrogenation at 325° C. in the presence of palladium catalyst, the so-called Puregas unit, has also been reported. The major effort for the quarter was, therefore, development of a suitable adsorber and determination of its capacity.

2.0 Summary

At the beginning of the quarter, work was undertaken to relocate the gas train used for removal from the gas stream of components which would interfere with adsorption of krypton and xenon. Also undertaken was construction of an adsorber with a refrigeration system based on the use of liquid nitrogen under pressure.

Unexpected difficulties in operation of the relocated gas train slowed experimental progress, but five adsorption tests were made. In the tests the gas mixtures containing krypton and xenon were passed through the adsorber and the concentration of krypton in the exhaust gas plotted as a function of time. It was established that the pattern of break-through closely followed theoretical considerations. Accordingly, rather precise predictions of the life of any such adsorber can be made provided concentration of the components in the inlet stream is known and a permissible value in the exhaust gas is established. Roughly





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2.0 Summary (Continued)

speaking, with a contact time of 10 seconds, each gram of carbon in the adsorber will adsorb approximately 50 standard cubic centimeters of krypton and xenon from a gas containing in the range of one-half per cent of these elements before the efficiency of the adsorber drops below 95%.

Considerable improvement in technique for determination of krypton concentration by counting krypton-85 tracer was achieved during the quarter. A commercial type RCL counter-tube was substituted for the special, gas-purged, gold-plated-rubber-hydrochloride-window tube. This development increased the reliability of determinations and made it possible to establish a precise pattern for breakdown of the adsorber.

A thermal decomposition procedure for decomposition of nitrous oxide has been developed. Essentially complete decomposition of nitrous oxide in a gas mixture was achieved by passing a gas mixture containing nitrous oxide through an Inconel pipe at 980°C. with an exposure time of 7 seconds.

3.0 Experimental

3.1 General Procedure

Off-gas from non-radive slug dissolution contains nitrogen, oxygen, carbon dioxide, carbon monoxide, water vapor, NO, NO2, N2O, and hydrogen. The gas mixture is collected over water in a gas holder. In the gas holder most of the NO, dissolves, forming nitric acid and nitrous acid. Any NO present may be oxidized to NO₂ by addition of oxygen and the NO₂ so formed is also allowed to dissolve in the water. Since "non-radive" slugs produce no krypton or xenon, it is necessary for experimental purposes to add known quantities of stable isotopes of these elements to the gas remaining after removal of the NO and NO2. Then, in order to determine the concentration of krypton at any point in the train, sufficient krypton-85 is added to act as a tracer. For an experiment, the pressure of the gas mixture is increased to approximately 27" water above atmospheric and the gas thereby driven through the gas train (Shown in Figure I) at a constant measured flow rate. The first unit of the train is a 20% KCH column which removes carbon dioxide and traces of NO2 remaining in the gas after treatment in the gas holder. Next the gas is mixed with hydrogen and passed through a palladium catalytic unit in order to remove oxygen and nitrous oxide. The quantity of hydrogen used is 50% excess over that theoretically required to convert the oxygen and NoO present to water and nitrogen. In order to prevent high concentrations of hydrogen, which might present an explosion hazard, the gas may be diluted with superheated steam during its passage through the catalytic unit. The gas is next cooled to condense out water vapor which is drained off through a liquid seal. The remaining gas is further dried, allegedly to a dew point of -40° or better by exposure to a bed of activated alumina. The dried gas is passed to a cold trap at -170° C. and finally to the carbon adsorber bed, also at -170° C. Exhaust gas from the adsorber is passed through a beta-gamma counter which measures its radioactivity. From the activity, as compared to the original gas, the concentration of krypton can be calculated.

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3.0 Experimental

3.1 General Procedure (Continued)

Since the concentration of krypton and xenon in the gas mixture is low a considerable quantity of gas mixture must be passed through the adsorber before significant break through occurs.

In order to reduce the labor and expense which would be involved in preparing and storing large quantities of dissolver off-gas, synthetic gas mixtures were used for adsorption tests. The synthetic mixtures approximated the concentrations of nitrogen, oxygen, and nitrous oxide which had been observed in the off-gas, but contained no NO or NO. Such synthetic mixtures could be prepared, spiked with xenon, krypton, and tracer, then kept under pressure in a storage tank of reasonable size.

3.2 Pure Gas Unit

Two attempts were made to conduct gas thru the relocated train using the "Pure gas" unit for removal of nitrous oxide and oxygen in the gas mixture. In both cases the "Pure gas" unit became plugged, resulting in failure of the experiments. In the first experiment the plugging occurred during the "warm up" period prior to actual admission of the gas. The unit was sawed open for examination. It was found that the catalyst pellets were cemented together into a solid mass and that a sample of the material dissolved in water was strongly basic, indicating entrainment of KCH solution from the caustic scrubbing column.

A check revealed that potassium hydroxide solution was being entrained, so the top of the column was modified and the de-entrainment trap was equipped with a continuous drain through a liquid seal. This served to indicate pressure in the train at that point as well as assuring removal of potassium hydroxide solution carried over from the column.

In spite of the precautions described, a new "Pure gas" unit plugged during the first two hours of use. In this case there was no evidence of caustic entrainment; it appeared that primarily the failure was due to corrosion of the "Poronze" filter at the exhaust end of the unit. Substitution of a filter of different material should prevent a recurrence of this type of failure.

3.3 Thermal Decomposition

At the beginning of the quarter, equipment was set up in order to develop a thermal decomposition method of removing nitrous oxide from the gas stream. The equipment was very simple, consisting merely of a 70 gallon galvanized steel tank in which gas mixtures containing nitrogen, oxygen, and nitrous oxide could be made up and dispensed through a pressure regulator, a throttling valve, and a gas rotameter to a tube furnace. By analysis of the exhaust gas from the furnace, it was possible to determine the effectiveness





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3.0 Experimental

3.3 Thermal Decomposition (Continued)

of nitrous oxide decomposition at various temperatures and flow rates. For preliminary experiments, a 5/8" diameter Inconel tube was used with a heated length of approximately 15 inches. With this apparatus it was established that approximately 7 seconds residence time at 980°C. was necessary in order to obtain essentially complete decomposition of nitrous oxide and that under these conditions approximately 10% of the nitrous oxide was converted to NO₂.

The equipment was scaled up to the size necessary for the flow rates used for the gas train and further testing conducted. Data from all experiments are grouped in Table I. It is felt that a more reliable method of nitrous oxide analysis must be achieved before the degree of decomposition as a function of time and temperature can be plotted closely, but the results indicate that decomposition under the conditions specified above are definitely quite complete. Since the difficulties experienced in operation of the "Pure gas" unit were holding up adsorption experiments, the new thermal decomposition unit was piped into the train ahead of the caustic scrubber and the "Pure gas" unit was by passed. Gas samples, taken during the adsorption experiments conducted since have afforded further assurance that the thermal decomposition unit satisfactorily removes nitrous oxide.

3.4 Counting

In previous work, counting of the radioactive gas was performed using a special counter tube. The construction of this counter tube, in brief, was such that it utilized a gold-plated rubber-hydrochloride window and required a continuous supply of counter gas.

Systematic experiments conducted during this quarter indicated that the operation of the tube was extremely erratic. Very slight changes in flow rate of gas caused large unpredictable changes in counting rate. Furthermore, the tube was rather insensitive. In view of these observations, experiments were undertaken to devise a more dependable and sensitive counter. It has developed that a type RCL counter-tube enclosed in a bottle through which gas can be passed is not affected by reasonable variations in flow rate of gas and, moreover, that this commercial counter is roughly 100 times as sensitive as the special counter tube used previously. A further advantage is that the commercial counter tube can be simply replaced by a new, standardized unit in the event of failure during operation.

3.5 Adsorption

Five adsorption test runs have been made in which gas mixtures containing known concentrations of krypton and xenon were passed through the adsorber at measured flow rates and break-through determined as a function of time. In all of the experiments the adsorber was a 35-gram bed of 4 to 6 mesh



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3.0 Experimental

3.5 Adsorption (Continued)

CXA Columbia carbon. The bed was 6 inches in length, 1.04 inches in diameter and was maintained at a temperature of -170° C. during operation by immersion in liquid nitrogen under a pressure of approximately 125 pounds per square inch. It was felt that an adsorber of smaller dimensions might lead to error due to faulty packing of the carbon pellets.

In experiments #1 and #2 the gas was passed through the gas train, using the thermal decomposition unit for removal of nitrous oxide. In experiments #3, #4, and #5, no nitrous oxide was added in the synthetic mixture and only the dryer, cold trap, and adsorber were operated. In all of the experiments the concentrations of nitrogen and oxygen in the gas mixture getting to the cold trap were approximately 89% and 10%, respectively.

Data from the experiments are plotted in Figures II and III as concentration of krypton breaking through the adsorber versus time. Also in Figures II and III are notes pertaining to flow rates and gas concentrations of particular experiments.

The theoretical curves for experiments #1, #3, #4 and #5, represented by dotted lines in Figures II and III, were construced in accordance with the equations prescribed by S. H. Jury in report ORNL C.F. 51-7-41, "Design of Percolators", using data from experiment #2 as a base. It appears that the equations admirably represent the experimental data and, accordingly, rather accurate predictions may be made as to the operating characteristics of this type of adsorber.

4.0 Equipment

4.1 Thermal Decomposition Unit

Modifications in the gas train with regard to its utilization with the hydrogenation unit have been minor. However, for the experiments on adsorption tests, the hydrogenation unit was removed and a thermal decomposition furnace placed ahead of the caustic scrubbing column as a substitute. This thermal decomposition unit is a 36" length of $l\frac{1}{2}$ inch schedule l_10 Inconel pipe which can be heated to l_1000 ° C. by two l_10 " tube furnaces. Power to the furnaces was supplied by a pair of 20 ampere Variacs which in turn were controlled by a single thermocouple and an indicating temperature controller. The wiring was arranged so that the voltage output of the Variacs was proportionally controlled, not completely cut on or off, by the temperature controller. The two l_10 " furnaces were separated by l_2 inches of insulation. This gave a temperature distribution along the heated pipe which had a pair of peaks with a slight dip between them. This served to lengthen the heated zone and flatten the extreme temperature peak which would normally be present near the midpoint of the heated zone.





4.0 Equipment

4.1 Thermal Decomposition Unit (Continued)

A graph of the temperature distribution versus length, together with a wiring diagram of the furnaces is shown in Figure IV.

4.2 Adsorber

The cold-trap and adsorber with their refrigeration unit is shown schematically in Figure V. The design is such that the unit operates continuously at -170° C. with a constant liquid level of nitrogen around the cold-trap and adsorber. Approximately 4 inches of hair-like insulation surrounds most of the refrigeration system and this is sealed from moisture by felt and creosote-base sealer.

The refrigeration unit operates at 125 pounds gauge pressure with liquid nitrogen as the refrigerant. As can be seen from the diagram, the upper reservoir can be isolated, bled to a atmospheric pressure, refilled, and brought back to operating pressure without affecting the lower reservoir which cools the cold-trap and adsorber. Consumption of liquid nitrogen by the refrigeration unit was about 4 to 6 liters per hour, but the unit could accomodate a much larger cold-trap and adsorber with practically no increase in consumption of liquid nitrogen. A similar unit has been designed for the Purex pilot plant which should not consume more than 10 liters of liquid nitrogen per hour but which has a carbon bed 40 times as large.

Many details have been omitted from Figure V for the sake of simplicity. However, it should be observed that high-pressure steam may be admitted to the system at D and condensate drained from E, enabling one to regenerate the adsorber without removing it from the refrigeration system.

TSM:mh

homas S. M. Mlan

TABLE I

Thermal Decomposition of Nitrous Oxide

Expt. Number		Concentr. Nitrous Oxide(%)	Temp.	Flow Rate (cc/min)	Contact Time (sec.)	Oxygen	st Concen Nitrous Oxide(%)	trations NO ₂ & NO	Percent N ₂ O Decomposed
17	5.7	7.7	800	1155			-		
2 T	5-7	7+7	800	697				-	
3 T	5.7	7.7	800	232		5.5	8.1	0.1	·
47	5.7	7•7	890	1155		5.7	7-3	0.2	-
5 T	5.7	7•7	890	691	1.5	5.7	6.9	0.2	10.4
6 T	5.7	7.7	890	198		5.8	7-4	0.5	
7 T	5.7	7.7	960	1164	0.89	6.9	4.4	0.6	43
8 T	5.7	7.7	960	705	1.47	7.7	2.4	0.9	69
9 T	5.7	7.7	960	699	1.49	7.4	2.9	0.6	62
10 T	5.7	7-7	960	176	5.9	8.7	0.8	0.0	100
11 9	5.6	8.4	960	1116	6.26	7.2	0.0	0.8	100
12 <u>/</u>	7•7	39.0	960	1065	6.4	18.3	0.8	3.1	98
13 T	5.7	7.7	960	142	7.34	8.4	0.3	0.6	96
· 11. P	7.7	39.0	960·	612	11.2	18.9	3.7	2.3	91 ?
15 P	7.7	39.0	960	467	14.6	16.6	12.9	1.6	67 ?
16 P	7.7	39.0	980	10/16	6.4	19.3	0.9	2.9	97
17 P	7.7	39.0	980	675	9.9	21.1	0.0	2.1	100
18 P	7.8	5.6	980	1050	10.5	10.1	0.0	0.4	100
19 P	9.2	7-3	980	906	12.2	9.8	0.0	0.6	100
20 P	9.0	34.7	980	906	12.2	20.3	0.0	1.2	100
21 P	5.7	6.0	980	770	14.3	8.9	0.0	0.5	100
22 P	7.7	39.0	980	300	22.4	6.8	0.0	1.5	100
23 T	5.7		1020	1178	0.9	6.5	0.8	3.8	51 ?
24 T	5.7	7.7	1020	728	1.4	7.3	1.2	4.6	40 3
25 T	5.7	7.7	1020	178	5.7	8.9	0.8	0.0	100

NOTE: (1) Nitrogen is the remaining component of gas mixtures (2) Temperatures were measured using an unshielded chromel-alumel

thermocouple

3. Experiments with symbol "Twen conducted using 5/6" tabe neactor.

Experiments with symbol "P" were conducted using 1/2" pipe neactor.

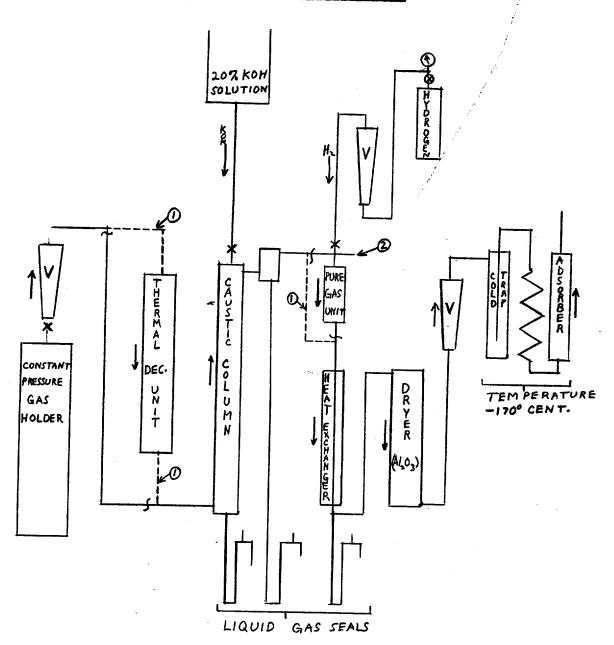
Experiments with symbol "P" were conducted asing 1/2" pipe neactor.





FIGURE I

Off-Gas Train



NOTES:

- 1. For operation with thermal decomposition unit flow follows dotted lines. By-passed units are indicated by "S"
- 2. During operation with "Pure gas" hydrogenation unit super heated steam is admitted at @ at a constant measured flow rate.

SECRET LEGONATION

HH

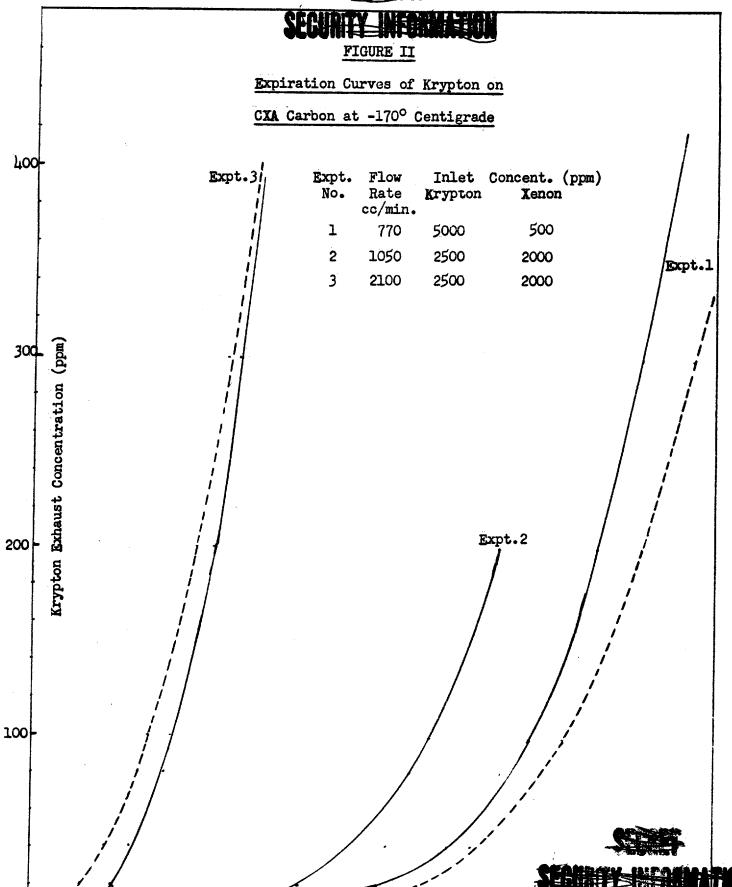


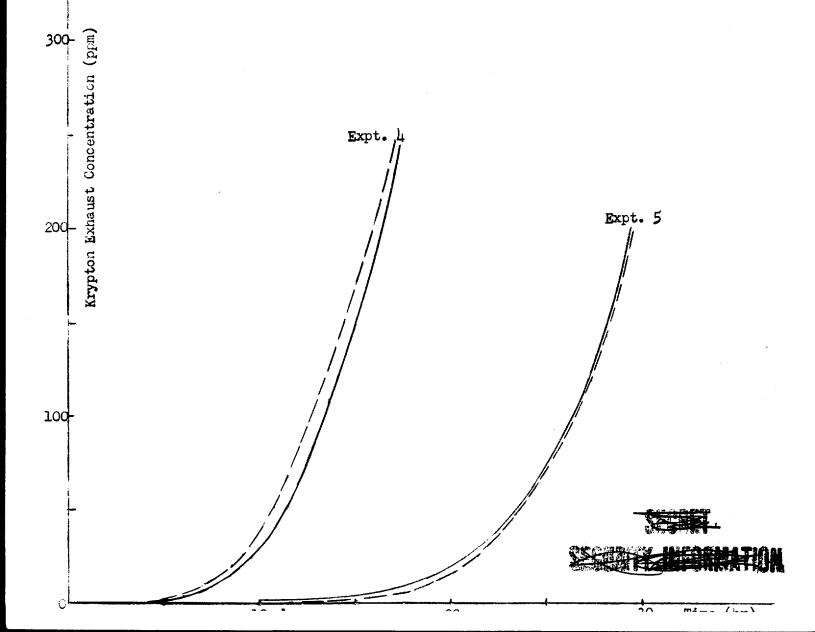


FIGURE III

Expiration Curves of Krypton on

CXA Carbon at -170° Centigrade

Expt. No.	Flow Rate cc/min.	Inlet Krypton	Concent. (ppm) Xenon
4	1050	1125	2000
5	533	2500	2000





Thermal Decomposition Unit

